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Characterisation and Resource Development of Steel Plant By-Product Streams

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Abstract
The recycling of steel plant by-products is a critical issue that needs thorough assessment and development for the sustainability of the integrated steel plant, from both environmental and economical perspectives. Research is being undertaken in the Steel Research Hub that aims to realise the value of by-products to the Australian steel industry and minimise the amount of by-product going to long term stockpile. As the first step, dust from basic oxygen steelmaking (BOS), a major steel plant by-product stream, is being characterised so that its potential use on plant or elsewhere can be assessed and evaluated. BOS dust contains components including iron, flux and zinc units, which have value on plant and elsewhere. Realising this value by recycling or other processing will help to minimise emissions and reduce costs by replacement of raw materials. However, some components, such as zinc, can limit the amount of possible recycling within the steel manufacturing process, causing both process and/or occupational health and safety issues. In this paper, characterisation of the BOS dust by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) analysis, X-ray diffraction (XRD), thermo-gravimetric analysis (TGA) and differential calorimetry (DSC) is presented and discussed with a view of understanding the behaviour of the materials in use and on plant and its limits for further processing.

Keywords
by-product, characterisation, resource, development, streams, plant, steel

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Characterisation and Resource Development of Steel Plant By-Product Streams

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Abstract: The recycling of steel plant by-products is a critical issue that needs thorough assessment and development for the sustainability of the integrated steel plant, from both environmental and economical perspectives. Research is being undertaken in the Steel Research Hub that aims to realise the value of by-products to the Australian steel industry and minimise the amount of by-product going to long term stockpile. As the first step, dust from basic oxygen steelmaking (BOS), a major steel plant by-product stream, is being characterised so that its potential use on plant or elsewhere can be assessed and evaluated. BOS dust contains components including iron, flux and zinc units, which have value on plant and elsewhere. Realising this value by recycling or other processing will help to minimise emissions and reduce costs by replacement of raw materials. However, some components, such as zinc, can limit the amount of possible recycling within the steel manufacturing process, causing both process and/or occupational health and safety issues. In this paper, characterisation of the BOS dust by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) analysis, X-ray diffraction (XRD), thermo-gravimetric analysis (TGA) and differential calorimetry (DSC) is presented and discussed with a view of understanding the behaviour of the materials in use and on plant and its limits for further processing.

Keywords: BOS steelmaking, by-product materials, integrated steelmaking, recycling, zinc.

1 Introduction

Research into on-plant recycling of by-products has been driven by the desire to improve the environmental and economic sustainability of the steel industry [1,2]. In the ARC Research Hub for Australian Steel Manufacturing (Steel Research Hub), a research program has been developed with the principal aims to realise the value in by-products and to minimise the amounts of potential by-products going to stockpiles.

Significant amounts of dust are formed in basic oxygen steelmaking (BOS), when oxygen blown at supersonic velocities impacts on the liquid steel. This dust is made up of droplets of liquid steel, slag and fine particulates from the added fluxes [1-4]. As steelmaking temperatures are often greater than 1600°C, elements such as zinc or lead can volatilise from the bath and enter the gas phase. As the gas phase is cooled, these volatilised metals can condense as separate particles or onto existing dust particles. Subsequent recycling of BOS dust back into the steelmaking route can therefore result in a recirculating load of zinc, which may lead to process issues. Knowledge of the form in which the iron and zinc are found within the BOS dust is crucial in understanding how to best recycle the dust or how best to process it for recycling [2,3,5]. This information also has application in understanding and developing novel ways to separate the zinc from the dust [5-9].

A well-known issue with steel plant by-products is their high variability [1,2,5,6]. In the case of BOS dust, the composition and particle size can change from heat to heat, depending on the target and hot

metal (and scrap) composition, as well as within a single heat, depending on the blowing conditions and any additions made (fluxes and coolants). This makes these materials difficult to use, both for characterisation at a laboratory scale and for recycling at the steel plant.

At BlueScope, the BOS dust is separated from the off gas by a wet scrubbing process, which is then dewatered into a filter cake using a tube press filter. This dewatered filter cake is stored in stockpiles prior to recycling. Experience has shown that the BOS filter cake stockpiles have a tendency to self-sinter. This self-sintering is likely a result of exothermic oxidation reactions. The sintered material is preferred for recycling as it has better physical properties (higher strength and larger particle size) compared to the original filter cake. This allows the sintered BOS filter cake to be directly recycled to the BOS as a coolant, replacing iron ore. Undersize self-sintered material is currently recycled to the sinter plant at a low rate, limited by the blast furnace zinc load limit for both process stability and occupational health and safety. Enhancing the in situ sintering/oxidation of the BOS filter cake will allow more material to be recycled to the BOS (decreasing the need for relatively expensive lump ore additions) and decrease the proportion being fed to the sinter plant. This should lead to a decrease in production costs while also improving the environmental performance of the plant.

Better understanding of the oxidation and sintering behaviour of the BOS filter cake will allow optimisation of this process and improved recyclability via the BOS coolant route. To achieve this, the BOS filter cake from BlueScope's Port Kembla steelworks has been characterised by several techniques, including scanning electron microscopy (SEM), X-ray diffraction (XRD), thermo-gravimetric analysis (TGA) and differential scanning calorimetry (DSC). Freshly produced BOS filter cake was characterised and compared to samples after processing in the TGA. In addition to characterising the phases present, morphology and oxidation behaviour of the BOS filter cake, a focus was also placed on the representation and repeatability of the characterisation.

2 Experimental

Characterisation of the oxidisation and sintering of the BOS filter cake during heating to 1000°C in air was carried out by heating samples in air using a large scale TGA and a small scale TGA-DSC. Samples before and after reaction were characterised by SEM and XRD.

BOS filter cake samples in the form of dense "biscuits" (Figure 1(a)), composed of dewatered BOS dust slurry were obtained prior to stockpiling and contained 13.2 mass% moisture as sampled. The dry composition of the BOS dust is given in Table 1.

Table 1: Composition of BOS filter cake, as measured by EDS (in mass%).

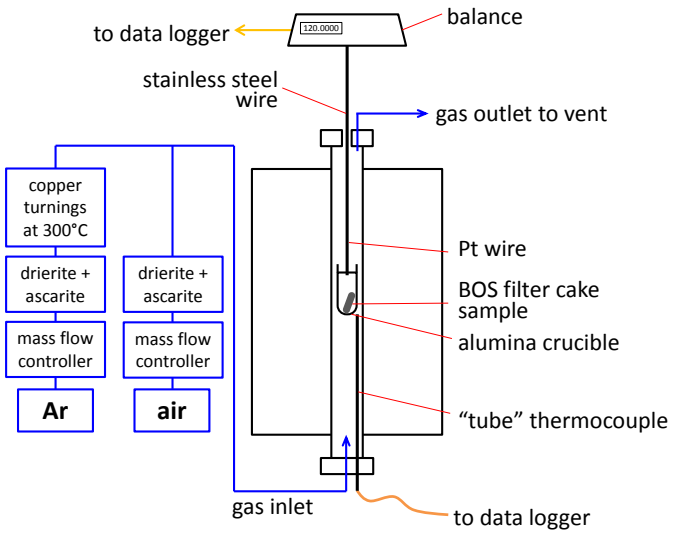
Total Fe	SiO_2	Al_2O_3	CaO	MgO	MnO	P	Zn	Pb
56.7	2.1	< 1	4.2	1.5	1.1	< 1	13.6	< 1

A large scale TGA system, as shown in Figure 1(b), was used to measure weight change during reaction of the BOS filter cake with air while heating to 1000°C. A single BOS filter cake "biscuit" with a nominal mass of 20 g was placed in an alumina crucible hung from the balance. The crucible had twelve 8 mm holes circumferentially around the bottom end of the crucible for gas access. In order to separate the drying and oxidation processes, the sample was held at 100°C under Ar flowing at 1 L/min for 70 minutes. The system was then heated at 10°C/min to 1000°C under air flowing at 1 L/min. The system was held at 1000°C in the flowing air for a further 30 minutes before cooling under Ar. The gases used were high purity (99.99%) and further purified by passing through drierite and ascarite prior to entering the furnace. The Ar was further cleaned by passing it through Cu turnings at 300°C.

The weight of the sample was logged during heating and the calculated fractional weight change (FWC) used to characterise the extent of reaction of the sample according to (1),

$$FWC = (W-W_o)/W_o \quad (1)$$

where W_o and W are the initial mass and the mass of a sample at time t, respectively in grams. The data were logged at a 1 s frequency.



(a) (b)

Figure 1: (a) BOS filter cake "biscuit"; (b) Schematic of large scale TGA set-up.

The thermal behaviour of the BOS dust was also measured using a Mettler Toledo Star^{e} TGA-DSC1. A BOS filter cake "biscuit" was crushed, and a nominal 100 mg sample was placed in an alumina crucible. The heating program in the TGA-DSC was different to that used in the large scale TGA, with the sample heated from room temperature to 1000°C at 10°C/min. A flow of air was maintained across the sample for the entirety of the experiment. The weight and temperature of the sample, and energy flow to the sample were logged at a 1 s frequency.

For both the large scale TGA and the TGA-DSC, at least three samples (from the same batch of BOS filter cake) were measured in order to help evaluate repeatability and sampling representation.

Samples were prepared for microscopy by mounting in epoxy resin, followed by sectioning, grinding and polishing to a 1 µm finish. These samples were examined using back-scattered electrons in an SEM, equipped with an energy dispersive spectroscopy (EDS) detector for elemental analysis. Samples for XRD were ground in a mortar and pestle. The diffraction patterns were collected using Cu-Kα radiation, with a Ni filter at 35 kV and 1 kW of power.

3 Results

Typical FWC curves for the reaction of the BOS filter cake with air during heating to 1000°C are shown in Figure 2. The TGA curves from both the large scale TGA (Figure 2(a)) and the smaller scale TGA-DSC (Figure 2(b)) have similar weight change characteristics, indicating similar reaction behaviour of the BOS filter cake in both techniques. This is despite the large difference in sample size and consequent thermal response of the two techniques. It is apparent that several different processes occurred at different temperatures during heating.

The rate of weight change was plotted against temperature to assist with determination the temperature ranges at which the different processes occurred. These curves were also used to help evaluate the repeatability of the test and representation of the samples. The rate against temperature plots for four repeats in the large scale TGA are given in Figure 3. It can be seen that the reaction of the BOS filter cake during heating follows a very characteristic behaviour. There are two sharp increases in the rate at 120-130°C and 730-770°C. In between these ranges, the rate of weight change decreases with temperature, crossing from weight gain to weight loss at 600-620°C. Characterisation of the reacted samples (detailed below) showed these increases in weight are likely due to oxidation of the BOS filter cake. These results demonstrate good repeatability between the larger ~20 g samples used in the large scale TGA. Similar repeatability was found for the smaller (~100 mg) samples used in the TGA-DSC.

From the behaviour seen in Figures 2 and 3, the characteristic behaviour of the BOS filter cake during heating can be separated into several distinct segments:

1. Drying of the sample at temperatures below ~110°C. This is represented by the large weight decrease.
2. Oxidation of the sample, beginning at ~120-130°C and complete at ~600°C, represented by the increase in the sample weight.
3. A slight decrease in sample weight between ~600-620°C and ~750°C.
4. A slight further weight gain caused by oxidation at temperatures between ~730-770°C and ~900°C.
5. A small decrease in sample weight at temperatures above 880°C.

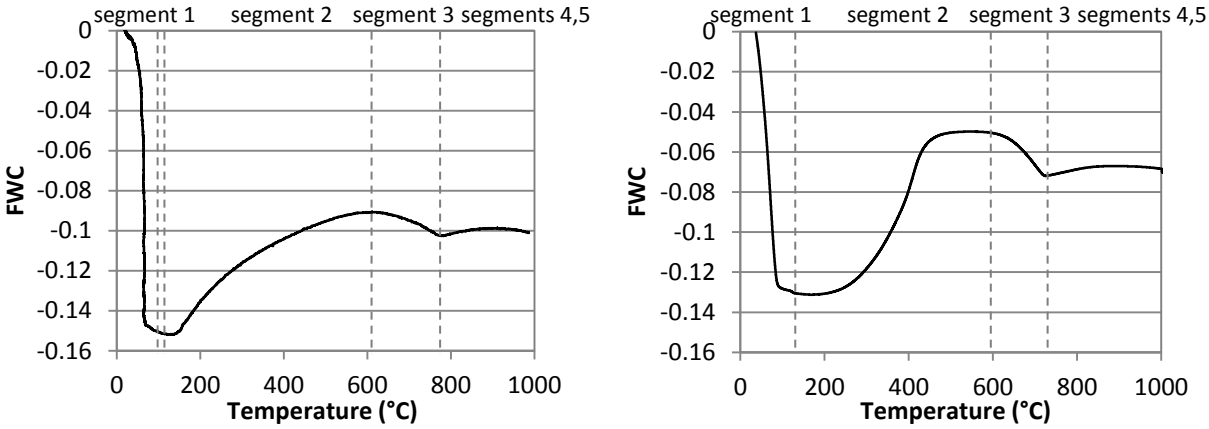


Figure 2: FWC curves for BOS filter cake oxidation in (a) the large scale TGA (20 g samples); and (b) the TGA-DSC (100 mg samples).

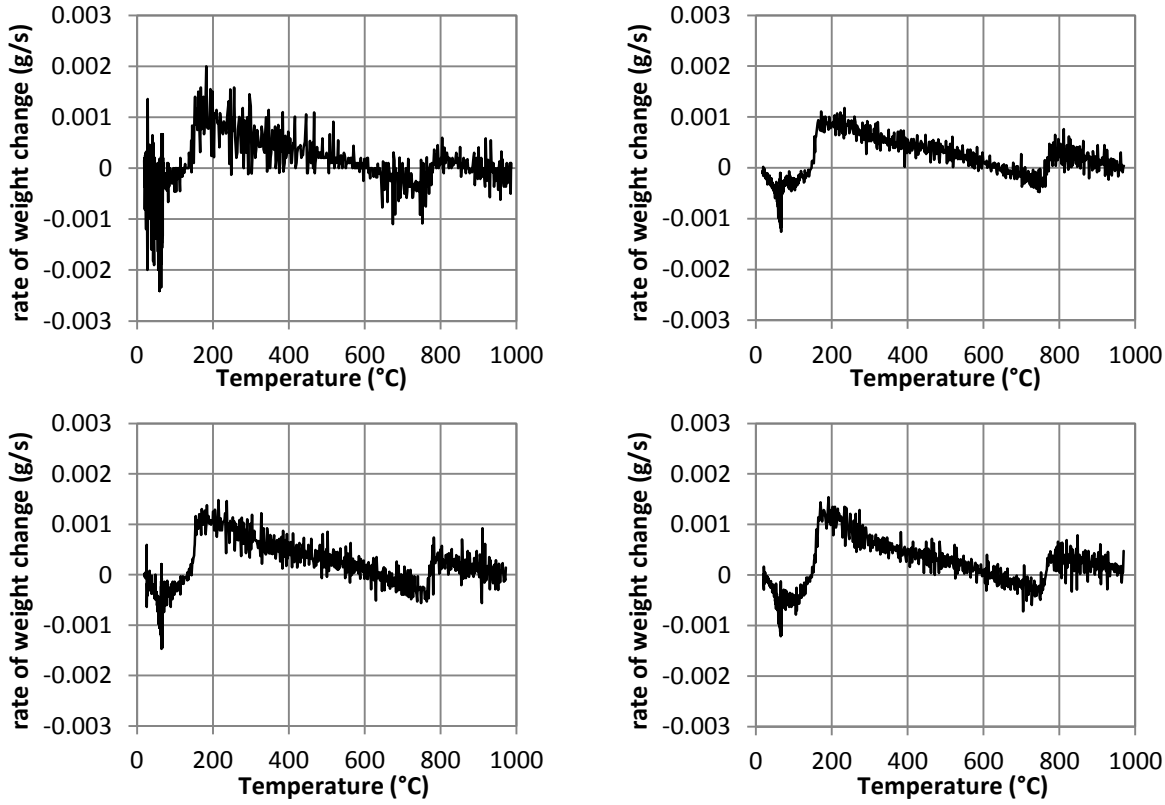


Figure 3: Rate of weight change plotted against temperature for BOS filter cake during oxidation during heating to 1000°C for four repeats in the large scale TGA.

The thermal behaviour of the BOS filter cake during oxidation was measured using DSC. A typical heat flux curve is given in Figure 4 which corresponds to the TGA measurement in Figure 2(b). Endothermic or exothermic behaviour can be seen as occurring below or above the fitted baseline, respectively. The thermal behaviour of the sample matches well with the events seen in the weight change curves. Endothermic drying of the sample occurs at low temperatures (<100°C) (segment 1). A large exothermic peak between ~150 and ~500°C corresponds to the oxidation of the BOS filter cake (segment 2). A much smaller exotherm can be seen above 710°C, which appears to correspond to both segments 4 and 5 above.

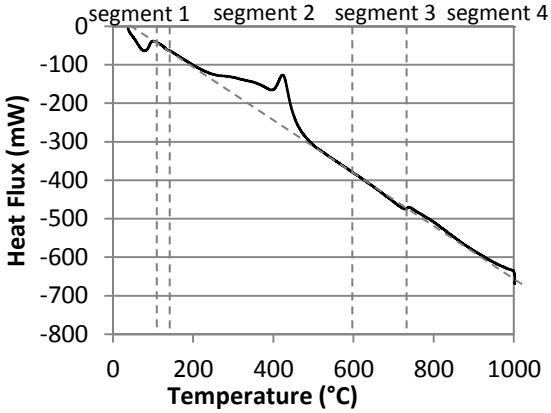


Figure 4: Heat flow curve from TGA-DSC for BOS filter cake being heated to 1000°C in air.

Phase analysis by XRD (Figure 5) of the fresh (unreacted) BOS filter cake shows that it predominantly contains metallic iron (Fe) and wüstite (FeO). Other components include smaller amounts of zinc ferrite ($ZnFe_2O_4$) or magnetite (Fe_3O_4), zincite (ZnO), fluxes (calcite, $CaCO_3$) and some graphite. It can be difficult to distinguish between magnetite and zinc ferrite by XRD as they have the same crystal structure and similar lattice parameters [10], and can form a mutual solid solution.

A typical XRD pattern of a post-reaction sample taken after the TGA test is also given in Figure 5. It can be seen that during reaction the sample was oxidised from the initial predominantly metallic iron and wüstite to hematite and magnetite-zinc ferrite. As the post-reaction sample appears to be predominantly hematite and zinc ferrite (both containing Fe^{3+}) the sample has been nearly fully oxidised during heating to 1000°C in air.

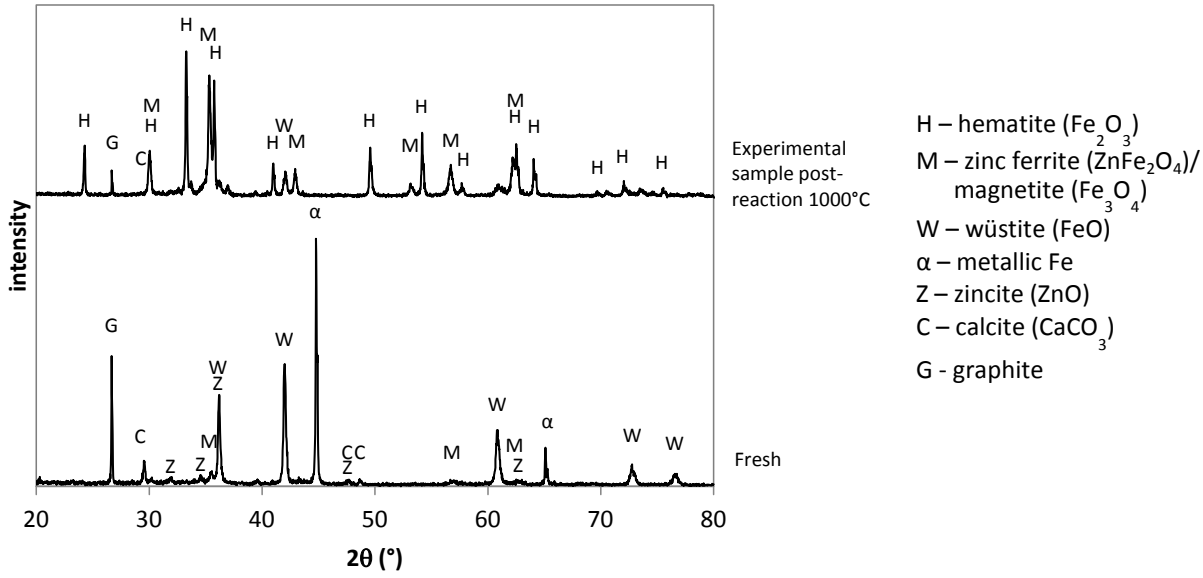


Figure 5: XRD patterns for BOS filter cake before and after oxidation during heating to 1000°C in air. The key for the peak labels is given on the right.

Micrographs of the fresh BOS filter cake are shown in Figure 6, while Figure 7 shows elemental mapping by EDS. The sample contained a matrix of fine metallic iron and iron oxides holding together larger particles of metallic iron, iron oxides, flux materials and some remnant slag droplets. The matrix of metallic iron and iron oxides was made up of very fine particles, typically 1 µm or smaller (Figure 6(b)). The larger particles were unevenly dispersed through the matrix. Remnant limestone particles were typically smaller (5-10 µm) than the dolomite particles, and were finely dispersed through the matrix phase. Zinc was generally evenly distributed through the matrix material. This well distributed zinc likely corresponds with $ZnFe_2O_4$ found in the XRD analysis. Some distinctly zinc rich particles, of a size approximately 1-2 µm can be seen in the Zn map in Fig. 7. These zinc-rich particles likely correspond to the small amount of ZnO identified in the XRD patterns.

The micrographs of the samples after reaction are shown in Figure 8. The microstructure has changed significantly from the fresh samples. At low magnification, densification of the structure can be seen, along with some interaction between matrix and the remnant flux and slag particles. Most obvious is the formation of a dense layer towards, but not at, the outer surface of the sample and near cracks. EDS analysis showed that this dense layer was Zn rich. At higher magnifications, the matrix can be seen to have sintered from the original discrete particles into a spongy structure reminiscent of porous refractories. There is little evidence remaining of the finely dispersed flux particles. Magnesium oxides were found in solution in the zinc ferrite. Calcium oxides were not found in the zinc ferrite phase, but rather as separate calcium ferrites.

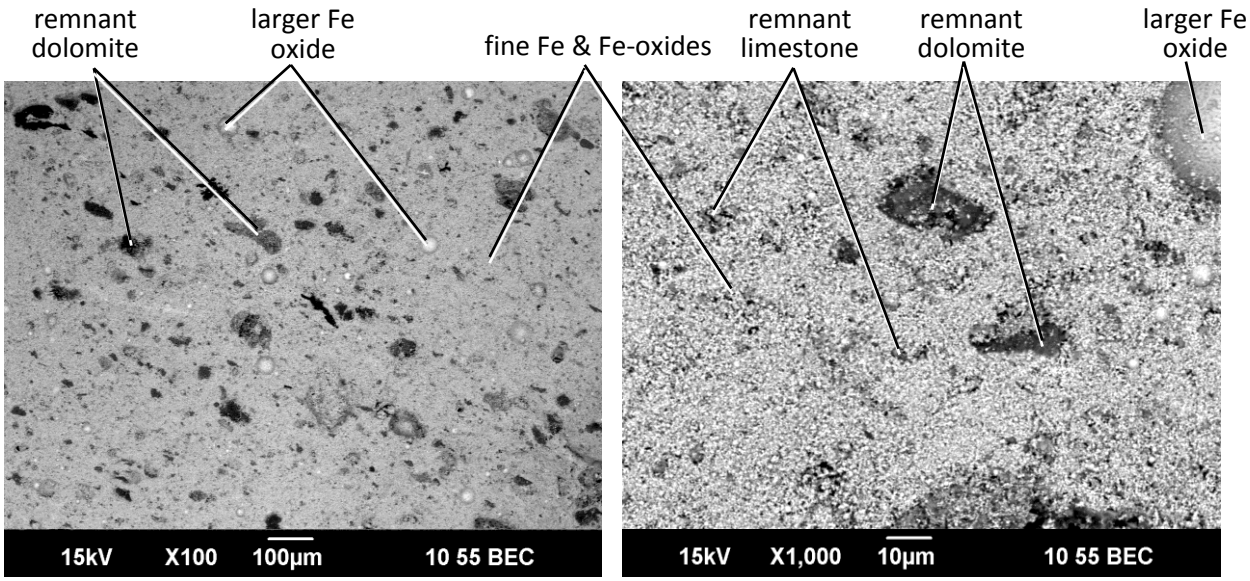


Figure 6: Back scattered electron micrographs of fresh BOS filter cake. (a) low magnification; and (b) high magnification.

4 Discussion

The phases present in the reacted BOS filter cake samples clearly show that the final sample is more oxidised than the initial material (Figure 5), as would be expected for a mixture of metallic iron and wüstite heated in air. This oxidation of the sample correlates with the increase in weight seen in the TGA curves and the exothermic peak seen in the DSC curve, indicating that the oxidation of the BOS dust appears to begin at low temperatures (~150°C) and is largely complete at 600°C. The micrographs show that the majority of the fresh BOS filter cake constituents are tightly packed, very fine particles of metallic iron and wüstite. Sub-micron particles of metallic iron would be expected to be highly reactive and susceptible to reaction at low temperatures. The processes occurring at higher temperatures (segments 3 and 4) have not yet been analysed, and will be the subject of future study.

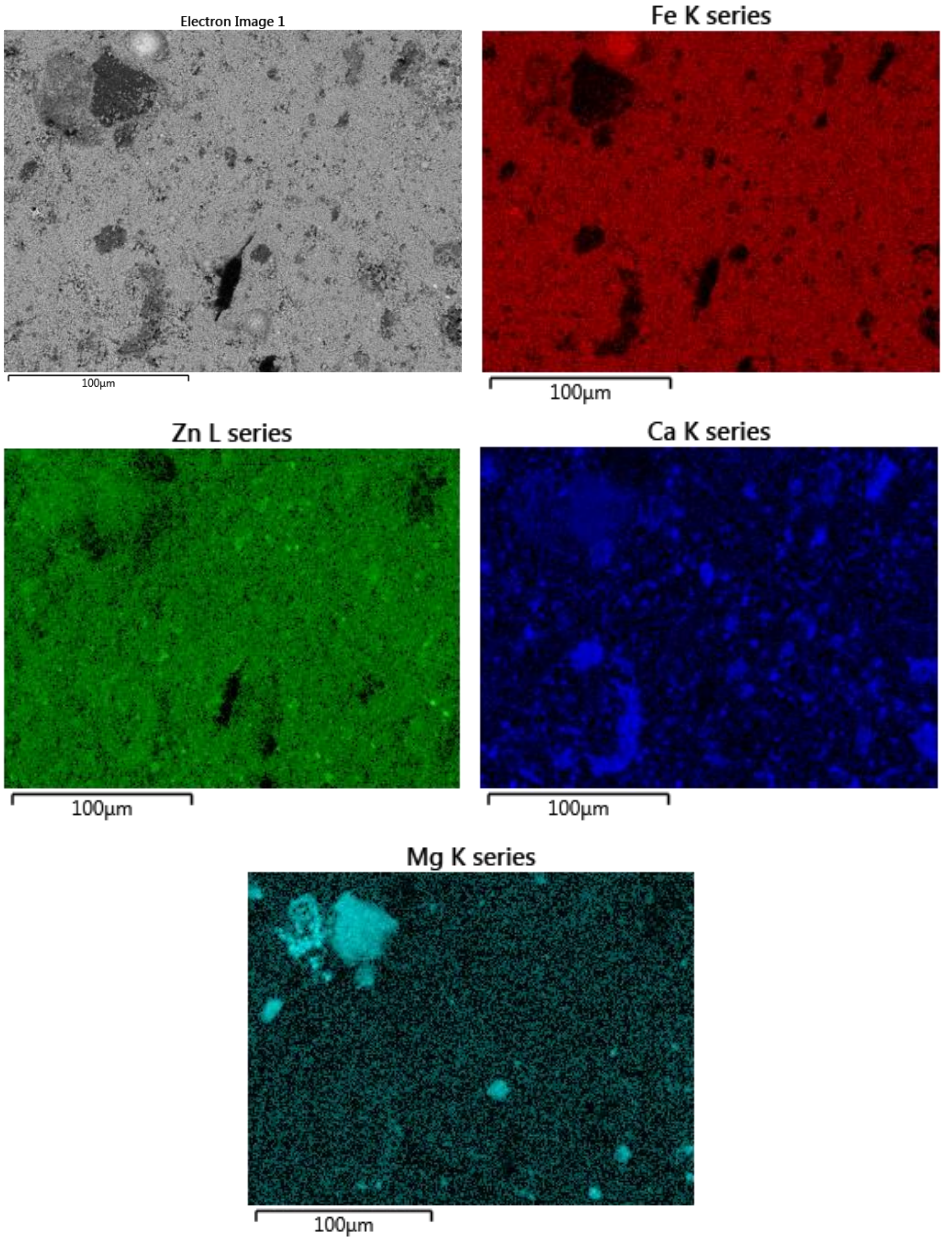


Figure 7: Elemental maps of Fe, Zn, Ca and Mg for a fresh BOS filter cake measured using SEM-EDS.

Based on the phases present in the BOS filter cake before and after reaction, several reactions are proposed in (2) to (5) that may represent the overall changes occurring during the heating of the BOS filter cake in air. Using FactSage [11], each of these reactions was found to be exothermic between 100-1000°C, matching what was seen in the DSC curves where only exothermic peaks were found. While reactions (2) to (5) represent the overall changes occurring, it is likely that they do not represent the reaction steps that occur during oxidation of the BOS filter cake.

$$2Fe_{(s)} + 1½\ O_{2(g)} = Fe_2O_{3(s)} \quad (2)$$

$$2FeO_{(s)} + ½O_{2(g)} = Fe_2O_{3(s)} \quad (3)$$

$$Fe_2O_{3(s)} + ZnO_{(s)} = ZnFe_2O_{4(s)} \quad (4)$$

$$2Fe_{(s)} + ZnO_{(s)} + 1½\ O_{2(g)} = ZnFe_2O_{4(s)} \quad (5)$$

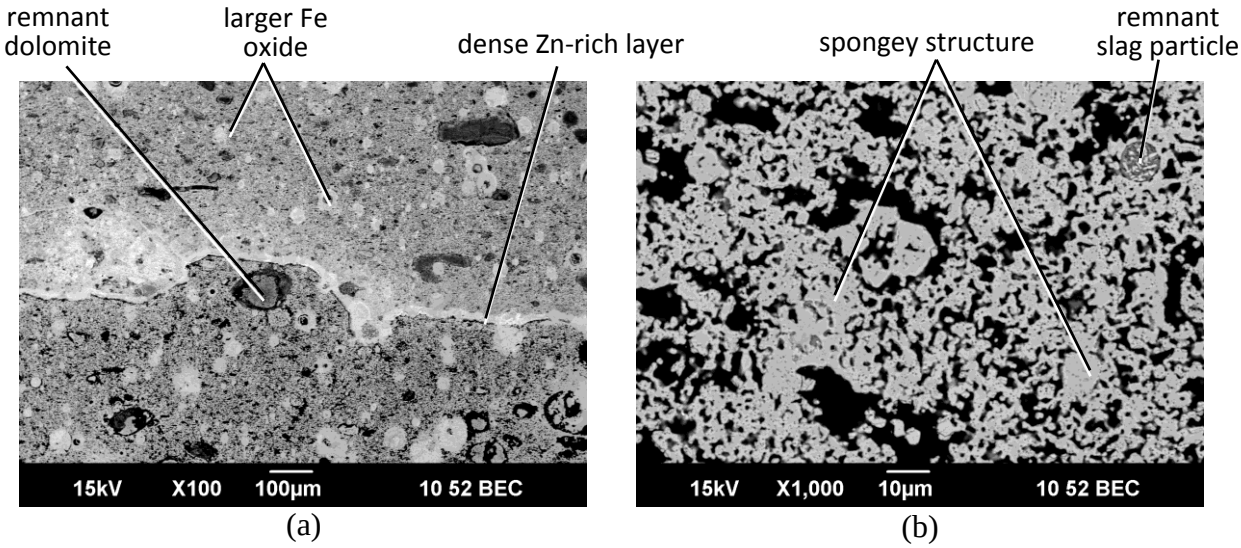


Figure 8: Back-scattered electron SEM micrographs of BOS filter cake after reaction in air during heating to 1000°C. (a) Low magnification image; (b) higher magnification image.

The phases present at equilibrium at 100 to 1000°C in air for a simplified Fe-Zn-O system with a composition corresponding to the unreacted BOS filter cake were predicted using FactSage [11]. At all temperatures, hematite (Fe_2O_3) and a spinel with a composition close to that for stoichiometric zinc ferrite ($ZnFe_2O_4$) were the only phases predicted. The results of this thermodynamic analysis show, as would be expected, that oxidation of the BOS filter cake is possible at low temperatures. The thermodynamic analysis predicts that all iron should be Fe^{3+}. The phases identified by XRD (Figure 5) are largely consistent with this. However, a small amount of wüstite was identified in the reacted samples, while it is also possible that some magnetite was also present (in solution with zinc ferrite). The presence of these Fe^{2+} containing phases is an indication that the sample was not at equilibrium and the oxidation of the BOS filter cake being kinetically limited. This is not surprising considering the short reaction times, and the large, dense samples used in the large scale TGA.

Zinc was mobile during the heating of the BOS filter cake to 1000°C, as seen by the formation of dense Zn-rich layers in Figures 8 and 9. XRD showed that all the zinc within the oxidised sample appeared to be in the form of zinc ferrite, with any zincite (ZnO) identified in the initial sample no longer present. Similar mobility of zinc was found by Wang et al. [12] who proposed a reduction-oxidation-nucleation based mechanism for this mobility.

The two different TGA set-ups used in this study gave slightly different results (Figure 2). These differences could be caused by two main factors. The thermal response of the two furnaces and the two different sample sizes cannot be expected to be the same. In the large scale TGA with the 20 g sample, it is also quite possible that temperature gradients are formed across the sample during heating. It is also probable that the mass transport of oxygen through the sample plays a larger role in determining the kinetics in the dense “biscuits” used in the large scale TGA than in the crushed samples used in the small scale TGA.

These represent some of the disadvantages of the large scale TGA. However, the 20 g sample size has the advantage of likely being more representative than the 100 mg samples used in the TGA-DSC. Figure 6(a) shows just how variable the BOS dust samples can be, even within a small cross-section of a single “biscuit”. While there will always be a question of whether the samples are representative of the bulk BOS filter cake stockpile, the 20 g samples, being two orders of magnitude larger than the 100 mg samples, will likely better represent the BOS filter cake.

With steel plant by-product materials like the BOS filter cake, there are significant concerns about sample representation and variability [1]. However, despite these concerns, it has been found that the results of the small scale TGA-DSC and especially the large scale TGA were consistent and repeatable (Figure 3). This gives confidence that the characteristic oxidation behaviour of the samples in the TGA tests reasonably represents the behaviour of the BOS filter cake during heating in air.

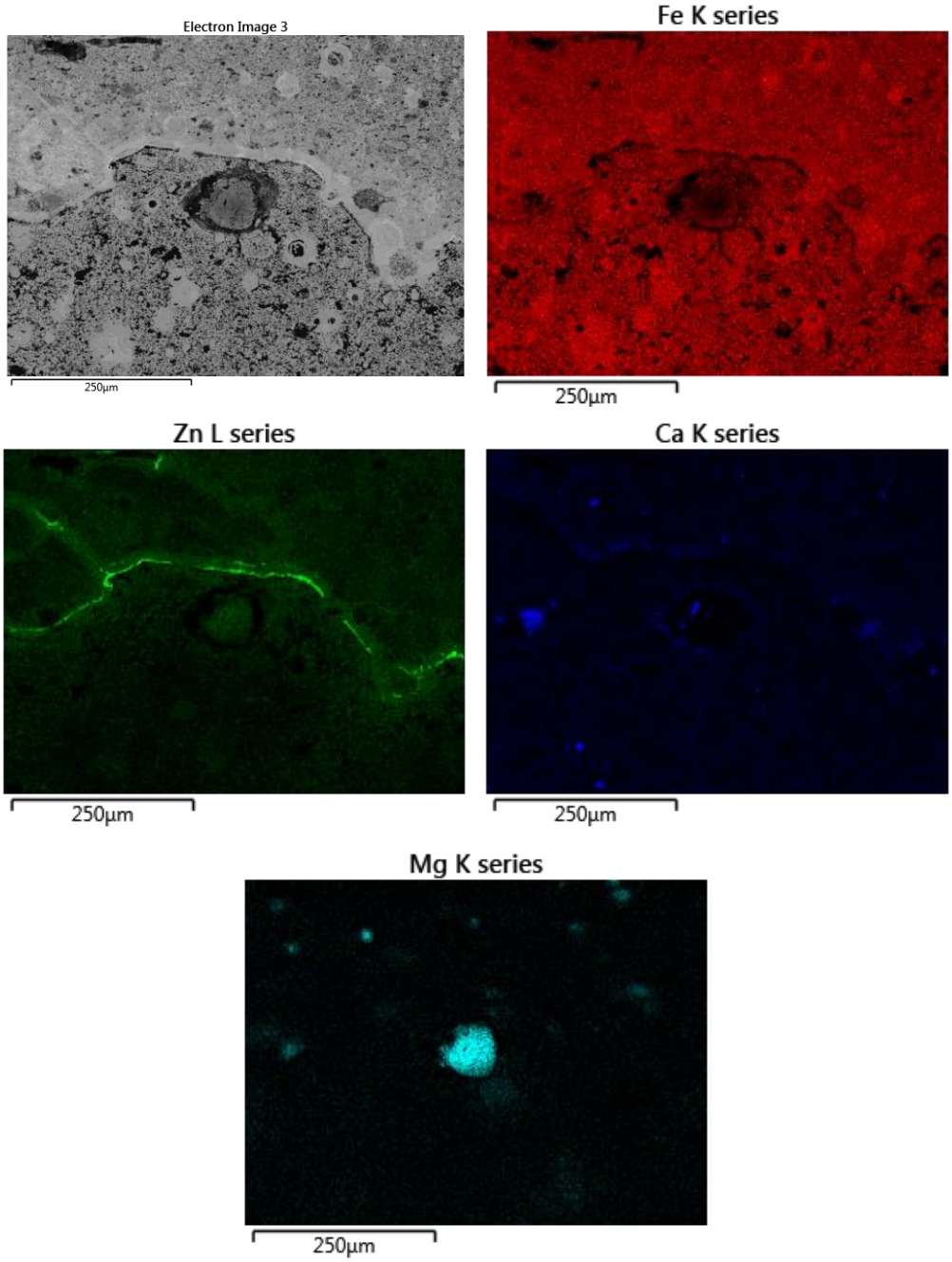


Figure 9: Elemental maps of Fe, Zn, Ca and Mg for a BOS filter cake sample after reaction with air during heating to 1000°C measured using SEM-EDS.

Linking the behaviour of the BOS filter cake in the TGA tests and the behaviour of the BOS filter cake in the on-plant stockpiles will lead to better understanding of the self-sintering. The onset of the reaction of BOS filter cake with air occurs once moisture has been removed from the sample. From Figures 2 and 3, it can be seen that the onset of weight gain (oxidation) occurs at temperatures as low as 120-130°C. This seemingly indicates that reaction of the BOS filter cake with air occurs in these experiments only after the moisture is removed. The BOS filter cake is stockpiled hot, at 50-70°C, which may provide enough heat to assist in locally drying the BOS filter cake. As the oxidation of wüstite and especially metallic iron in air are exothermic, any reaction occurring within the stockpile will serve to increase the temperature, further driving oxidation and increases in temperature.

Identified the characteristic weight changes that occur during oxidation of the BOS filter cake is a first step in the analysis of the processes occurring during self-sintering. Fully understanding these

processes, and what initiates reaction below 100°C and elevates the temperature of the BOS filter cake to the onset temperature for reaction with air at 120-130°C will be the topic for further studies.

Hence, likely factors that may possibly be controlled to promote self-sintering (and recycling) include the temperature of the BOS filter cake during stockpiling, the moisture level of the BOS dust and possibly the amount of metallic iron in the dust. While it cannot be controlled, the fine particulate nature of the metallic iron and wüstite in the BOS filter cake gives it the high reactivity required for the low temperature oxidation. Increasing the initial temperature, and decreasing the moisture content may promote the self-sintering of the BOS filter cake in the stockpiles, allowing more of the BOS dust by-product to be recycled back to the BOS as coolant.

5 Conclusions

A by-product from BlueScope's Port Kembla steelworks, BOS filter cake, has been characterised in terms of its behaviour during heating to 1000°C in air to better understand its self-sintering behaviour.

Characteristic behaviour of the BOS filter cake during heating in air to 1000°C was identified as being a complex, multi-step process, involving sequential drying and oxidation of metallic iron and wüstite, as well as the formation of zinc ferrite and reaction with fluxes. The onset of oxidation occurred after the moisture was removed from the BOS filter cake, at temperatures as low as 120-130°C.

Unreacted BOS filter cake predominately consisted of very fine (< 1 µm) particles of metallic iron and wüstite. The fine particulate nature of the BOS filter cake leads to it being highly reactive, even at low temperatures. After reaction with air at 1000°C, the BOS filter cake had predominantly transformed to hematite and zinc ferrite. The densely packed, fine particles in the unreacted filter cake had transformed to a porous, spongy morphology.

Several factors that were identified as likely being important, based on the results of this study, for the promotion of BOS self-sintering in stockpiles. These included the elevated temperature at which the BOS filter cake is stockpiled, the fine particulate nature of the BOS dust and the level of moisture within the BOS filter cake.

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Raymond Longbottom has been a Research Fellow at the University of Wollongong since 2008, and within the Steel Research Hub since 2015. He studied at UNSW, completing his B.Met.E. in 2000, and his Ph.D. in 2005. Previously, he worked at the Norwegian University of Science and Technology (NTNU) as part of the EU-funded Ultra-Low CO_2 Steel (ULCOS) project. His interests lie in sustainable iron and steelmaking, in both conventional and alternative processing.